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AGRICULTURAL RESEARCH ADMINISTRATION

u.s. BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY

THE PHYSICAL PROPERTIES OF FATTY ACIDS, GLYCERIDES, AND DERIVATIVES

THE TECHNICAL LITERATURE AND PATENTS OF THE

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Issued: August 31, 1953

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INTRODUCTION

Research work on vegetable oils has been underway at the Southern Regional Research Laboratory of the U. S. Department of Agriculture since 1941. As a consequence of the research program on fats and oils, such products as "tailor-made" or modified fats, purified mono-, di-, and triglycerides and fatty acids, oils from minor oilseed crops such as sesame and rice, and products derived from these materials were prepared. Many of these natural and derived fatty materials were characterized with respect to their more important physical properties, such as solubility, expansibility, viscosity, density, specific volume, surface and interfacial tension, crystal structure, polymorphism, and spectra.

Special emphasis was placed on physical properties because they enter as basic considerations in most phases of fat and oil technology. Not only are these properties important in the design of equipment and the development of processes, but a large portion of the fat and oil industry is devoted primarily to the altering of the physical properties of fatty products to meet consumer needs.

This bibliography is a listing of the more important articles relating to physical properties of fatty materials. It is compiled for the convenience of research workers and processors engaged in investigating and producing vegetable oil products, and derivatives of vegetable oils. These abstracts were taken for the most part from Southern Regional Research Laboratory lists of publications and patents, AIC-188 and supplements, 5 unnumbered lists, and AIC-319 and supplements 1 and 2.

EXPANSIBILITY

Bailey, A. E. and Kraemer, E. A.
DILATOMETRIC INVESTIGATIONS OF FATS. I. APPARATUS AND TECHNIQUES FOR FAT DILATOMETRY.
Oil & Soap 21, 251-253 (1944).

A method for employing dilatometry, alone and in conjunction with calorimetric methods, for investigating phase changes in fats and oils over the partial or complete melting range of these materials is presented. By this method, any irregularities introduced by the appearance of polymorphic transformations are easily recognized.

Bailey, A. E. and Singleton, W. S.
DILATOMETRIC INVESTIGATIONS OF FATS. III. THE DENSITY EXPANSIBILITY, AND MELTING DILATION OF SOME SIMPLE TRIGLYCERIDES AND OTHER FATS.
Oil & Soap 22, 265-271 (1945).

Information is given on the expansibility of fats in the solid state and on the volume changes accompanying melting and polymorphic transformations. Dilatometric curves between about -38°C. and temperatures representing the liquid state were determined for a number of pure triglycerides and some commercial fats. From data on the liquid samples, determinations were made on density in both the liquid and the solid states, on expansibility of each state with increased temperature, and on the dilation accompanying melting. Volume changes accompanying the transformation from one polymorphic form to another were determined. Certain relationships are pointed out among the properties discussed.

Kraemer, E. A. and Bailey, A. E.
DILATOMETRIC INVESTIGATIONS OF FATS. II. DILATOMETRIC BEHAVIOR OF SOME PLASTIC FATS BETWEEN 0°C. AND THEIR MELTING POINTS.
Oil & Soap 21, 254-256 (1944).

Dilatometric curves between 0°C. and their melting points have been obtained for lard, butterfat, cottonseed oil, peanut oil, a commercial margarine oil, a commercial all-hydrogenated vegetable shortening, three samples of soybean oil hydrogenated to different degrees, a hard butter fractionally crystallized from hydrogenated peanut oil, a mixture of tristearin and peanut oil, and a synthetic fat composed of stearic and oleic acids. As a result of the interpretation of the dilatometric curves, the application of dilatometry to the qualitative and quanitative determination of glyceride composition in fats is suggested.

Singleton, W. S. and Bailey, A. E.
DILATOMETRIC INVESTIGATIONS OF FATS. IV. ESTIMATION OF SOLIDS AND LIQUID IN SOME PLASTIC FATS.
Oil & Soap 22, 295-299 (1945).

Dilatometric curves were determined over the complete melting range for samples of prime steam lard, oleo oil, all-hydrogenated vegetable shortening, and partially hydrogenated cottonseed oil, and estimates were made of the percentages of solids and liquid in the fats at different temperatures. The consistency of the fats, as measured by micropenetrations, was different for different fats which contained equal percentages of solids. The consistency, therefore, is not determined solely by the amount of solids present, but also by the character of the solid particles.

Singleton, W. S. and Gros, Audrey T.
DILATOMETRIC INVESTIGATIONS OF FATS. VI. MELTING DILATION AS A FUNCTION OF CHAIN LENGTH IN FATTY ACIDS AND THEIR GLYCERYL ESTERS.
J. Am. Oil Chemists' Soc. 29, 149-151 (1952).

Melting dilations were determined for the even-numbered, saturated n-fatty acids in the series lauric-stearic, and some of their glyceryl esters. These data are correlated with previous data to determine the relationship between melting dilation and the effective chain length of these compounds. The melting dilations of the mono-, di-, and triglyceryl esters were found to be proportional to the acid residue content of each compound. The melting dilation of a monoglyceride was one-half that of the diglyceride and one-third that of the triglyceride of the same acid. Equations were developed for calculating the melting dilation of the n-fatty acids and their glycerides as a function of chain length.

Singleton, W. S., O'Connor, R. T., Murray, Mildred and Pack, F. C.
DILATOMETRIC INVESTIGATIONS OF FATS. VII. MELTING DILATION AND POLYMORPHISM OF AN ALPHA AND BETA TUNG OIL.
J. Am. Oil Chemists' Soc. 29, 452-454 (1952).

The expansibilities of alpha tung oil and the three polymorphic forms of beta tung oil are determined for both the solid and liquid states, and the melting dilation of each substance calculated. The absolute densities are determined, and the absolute specific volumes are calculated. The content of solid and liquid phases over the ranges of melting of these oils were calculated. The isomerization of alpha to beta tung oil is accompanied by a volume decrease of 4%.

Singleton, W. S. and Vicknair, E. J.
DILATOMETRIC INVESTIGATIONS OF FATS. V. VOLUME CHANGES ASSOCIATED WITH THE POLYMORPHISM AND MELTING OF MONOSTEARIN.
J. Am. Oil Chemists' Soc. 28, 342-343 (1951).

The expansibility of the solid state, specific volume, and melting dilation of the different forms of 1-monostearin are, respectively, B-form 0.000241 ml./g./°C., 0.9419 ml./g., and 0.1367 ml./g.; B'-form 0.000291, 1.0307, and 0.0610; sub a-form 0.000303, 0.9515, and 0.1292. The melting dilation of stearic acid, monostearin, and tristearin were in direct proportion to mole percentages of stearyl groups in each compound.

Singleton, W. S., Ward, T. L. and Dollear, F. G.
PHYSICAL PROPERTIES OF FATTY ACIDS. I. SOME DILATOMETRIC AND THERMAL PROPERTIES OF STEARIC ACID IN TWO POLYMORPHIC FORMS.
J. Am. Oil Chemists' Soc. 27, 143-146 (1950).

The specific volume, expansion in both solid and liquid states, melting dilation, specific heat in both solid and liquid states, heat of fusion, and entropy of purified stearic acid (freezing point 69.20°C., melting point 69.5°C.) in both B- and C-forms were calculated from dilatometric and calorimetric data. The irreversible transition of stearic acid from the B- to the C-form was rapid and visible at 52.9°C. but also occurred at 35.2°C. when sufficient time was allowed for the transition to occur. The form of stearic acid was found to be dependent upon the temperature at which crystallization occurred. Crystallization at temperatures below the transition point obtained the B-form, and above the transition point only C-form of the acid was obtained, irrespective of the solvent used for crystallization.

Ward, T. L. and Singleton, W. S.
PHYSICAL PROPERTIES OF FATTY ACIDS. II. SOME DILATOMETRIC AND THERMAL PROPERTIES OF PALMITIC ACID.
J. Phys. Chem. 56, 696-698 (1952).

Pure palmitic acid has been examined by dilatometric and calorimetric methods. The properties investigated and the values found were (a) expansion of the acid in the solid and liquid states, 0.000280 and 0.000968 ml./g./°C., respectively; (b) melting dilation, 0.1806 ml./g.; (c) specific volume over the range of temperature from solid to liquid; specific heat; (d) heat of fusion, 51.2 cals./g.; and entropy.

Equations were developed for expressing the specific heat ($\underline{Cp}$) of palmitic acid at any temperature (°C.):

(solid state) $\underline{Cp} = 0.3831 + 0.0013\underline{t}$
(liquid state) $\underline{Cp} = 0.4624 + 0.00175\underline{t}$

INTERFACIAL TENSION

Feuge, R. O.

INTERFACIAL TENSION OF OIL-WATER SYSTEMS CONTAINING TECHNICAL MONO- AND DIGLYCERIDES.

J. Am. Oil Chemists' Soc. 24, 49-52 (1947).

Technical mono- and diglyceride products have been prepared from cottonseed oil-stearine, and the effectiveness of these products in lowering the interfacial tension at vegetable oil-water interfaces has been investigated. When both monoglycerides and diglycerides are present in the oil phase, the surface activity of the diglycerides is less than 1/100th that of the monoglycerides on a weight for weight basis; hence the interfacial tension is substantially a function of the monoglyceride content. The interfacial tensions against water of such oils as peanut, cottonseed, and soybean are practically the same, and are lowered by practically equal amounts for equal additions of a given monoglyceride preparation. A concentration of 1% of monoglycerides in the oil phase will lower the interfacial tension at the oil-water interface to approximately one-half that observed when no monoglycerides are present. A concentration of 6% of monoglycerides in the oil phase will lower the interfacial tension approximately to zero.

Gros, Audrey T. and Feuge, R. O.

MODIFICATION OF VEGETABLE OILS. X. EFFECT OF MONOGLYCERIDES ON THE INTERFACIAL TENSION OF OIL-WATER SYSTEMS.

J. Am. Oil Chemists' Soc. 28, 1-4 (1951).

The interfacial tension of mineral oil-, cottonseed oil-, and amyl acetate-water systems containing monoglycerides of oleic and linoleic acids, and saturated fatty acids from C_8 to C_{18} are determined. The ability of a given monoglyceride to reduce interfacial tension varied widely with the nonaqueous phase. On a molar basis the monoglycerides were equally effective with cottonseed oil and water; with mineral oil-water, monostearin was more effective. The effect of temperature on interfacial tension is related to the concentration of the glycerides.

Gros, Audrey T. and Feuge, R. O.

SURFACE AND INTERFACIAL TENSIONS, VISCOSITIES, AND OTHER PHYSICAL PROPERTIES OF SOME n-ALIPHATIC ACIDS AND THEIR METHYL AND ETHYL ESTERS.

J. Am. Oil Chemists' Soc. 29, 313-317 (1952).

Determinations of surface tension, interfacial tension against water, viscosity, density, and refractive index were made at 75°C. for the saturated, even-numbered n-fatty acids from $C_2$to C_{18} and for their methyl and ethyl esters. The effect of temperature on the physical constants was demonstrated for myristic acid. Surface tensions of the acids were in the range

19.6 to 27.7 dynes/cm.; the ethyl esters were about 2.5 dynes/cm. lower than those for the acids, and the methyl esters about 2 dynes/cm. lower than the acids. The viscosities increased as the number of carbon atoms increased; the densities decreased with increasing chain length; the refractive index increased with increasing chain length.

PLASTICITY

Feuge, R. O. and Bailey, A. E.
MEASUREMENT OF THE CONSISTENCY OF PLASTIC VEGETABLE FATS; A STANDARD MICROPENETRATION TECHNIQUE.
Oil & Soap 21, 78-84 (1944).

An improved micropenetrometer is described, for measuring the consistency of fats with a high degree of precision. The influence of various factors on the consistency of solidified fats has been investigated, and a standard technique for micropenetration tests is proposed. Micropenetration data are recorded on cottonseed, peanut, and soybean oils hydrogenated to different degrees, on blends of hydrogenated and unhydrogenated oils, and on commercial shortenings.

Feuge, R. O., Vicknair, E. J. and Lovegren, N. V.
MODIFICATION OF VEGETABLE OILS. XII. PLASTICITY OF SOME ACETO DERIVATIVES OF MONOSTEARIN.
J. Am. Oil Chemists' Soc. 29, 11-14 (1952).

Acetostearins were prepared, and their flexibility and melting characteristic determined. These materials were found capable of stretching more than 800% under the test conditions, and to possess good flexibility even at 4°C. The melting characteristics of the products were dependent on the purity of the monostearin used and the degree of acetylation. Some products melted completely below body temperature (37°C.) and others melted up to 60°. The temperature interval over which melting occurred was usually very short (1-3°)); however, products made from technical monostearin melted over a temperature range of 10-20°.

Kraemer, E. A., Smith, B. A. and Bailey, A. E.
MODIFICATION OF VEGETABLE OILS. II. A COCOA BUTTER SUBSTITUTE FROM COTTONSEED OIL.
Oil & Soap 20, 235-240 (1943).

Selective hydrogenation and solvent fractionation is used to prepare from cottonseed oil a hard butter very similar to cocoa butter, differing by the content of iso-oleic acid. Examination by micropenetration, solidification test, and dilatometry revealed minor physical differences. The new product exhibits the phenomenon of polymorphism to a less pronounced degree than cocoa butter.

SOLUBILITY

Bailey, A. E., Feuge, R. O., Kraemer, E. A. and Bauer, S. T.
MODIFICATION OF VEGETABLE OILS. I. NEW PRODUCTS BY FRACTIONAL CRYSTALLIZATION OF GLYCERIDES FROM PETROLEUM NAPHTHA.
Oil & Soap 20, 129-132 (1943).

A solvent process for the production of winterized cottonseed and peanut oils by low-temperature fractional crystallization is demonstrated to be feasible for the production of new products from cottonseed or peanut oils. The process is suitable for commercial use, since low ratios of solvent to oil are used, crystallization is at moderately low temperatures, and the solvent is easily obtainable in large quantities at reasonable price.

Bailey, A. E., Singleton, W. S. and Feuge, R. O.
PHASE INVESTIGATIONS OF FATS. I. APPARATUS AND TECHNIQUES FOR FAT-SOLVENT SYSTEMS AT LOW TEMPERATURES.
Oil & Soap 23, 201-204 (1946).

An apparatus that has been successfully employed for equilibrating and separating mixtures of fats and volatile solvents at relatively low temperatures is described, together with the technique for its manipulation. It consists of a constant-temperature bath controlled by on-and-off operation of a circulating pump through an electronic relay circuit, with a special toluene-filled thermoregulator, used in conjunction with an equilibrium-filtration assembly. The latter, consisting of a Pyrex glass apparatus equipped with a stirrer coil, clamped in a support of rigid copper tubing, with motive power for stirring the material furnished by an electromagnet held in a frame of rigid copper tubing, is so designed that it can be completely immersed in the bath liquid. There are no projecting parts to transmit heat to the material and the entire operation of equilibration and filtration can be accomplished without removing any part of the apparatus from the bath. With proper manipulation, losses of material are small. Two methods of calculating the composition of the solid and liquid phases are illustrated by data obtained in a typical experiment performed on the system stearic acid-oleic acid-commercial hexane.

Boucher, Richard E. and Skau, Evald L.
PHASE RELATIONS IN THE SOLVENT WINTERIZATION OF COTTONSEED OIL IN 85-15 ACETONE-HEXANE MIXTURE AT REDUCED HOLDING-TIMES.
J. Am. Oil Chemists' Soc. 29, 455-456 (1952).

Systematic winterization data are presented for different cottonseed oil-solvent mixtures and temperatures at different holding times. The results indicate that the holding time can be reduced from three hours to one hour by using either a higher oil concentration, a lower chilling temperature or both. The rate of crystal formation is increased by agitation.

Boucher, Richard E. and Skau, Evald L.
PHASE RELATIONS IN THE SOLVENT WINTERIZATION OF MOLECULARLY REARRANGED PEANUT OIL AND COTTONSEED OIL.
J. Am. Oil Chemists' Soc. 29, 382-385 (1952).
Phase-relation data were obtained to determine the effect of either mild or extensive molecular rearrangement on the winterization behavior of peanut and cottonseed oils. Results show that molecular rearrangement is disadvantageous, since a larger percentage of solid must be removed to obtain a winterized oil. In addition, the settling qualities of the solid are markedly impaired.

Boucher, Richard E. and Skau, Evald L.
PHASE RELATIONS PERTAINING TO THE SOLVENT WINTERIZATION OF PEANUT OIL IN ACETONE-HEXANE MIXTURES.
J. Am. Oil Chemists' Soc. 28, 501-504 (1951).
Phase relation data pertaining to the solvent-winterization behavior of a refined peanut oil in a mixed solvent consisting of 85% acetone and 15% commercial hexane, weight basis, are presented. Graphs are constructed to show the effect of oil-solvent ratio, chilling temperature, holding time, and agitation on the percentage of solid removed, on the degree of winterization, and on the settling qualities of the solid separating.

Boucher, Richard E. and Skau, Evald L.
PHASE RELATIONS PERTAINING TO THE SOLVENT WINTERIZATION OF COTTONSEED OIL IN HEXANE AND IN ACETONE-HEXANE MIXTURES.
J. Am. Oil Chemists' Soc. 28, 483-487 (1951).
Phase relation data pertaining to the solvent-winterization behavior of a refined cottonseed oil have been obtained for commercial hexane and a weight mixture of 85% acetone and 15% commercial hexane. Graphs are constructed to show the effect of oil-solvent ratio, chilling temperature, holding time, and agitation on the percentage of solid removed. The degree of winterization and the settling qualities of the solid separating. These data afford a basis for the selection of optimum conditions and procedures in the application of solvent winterization to cottonseed oil.

Fan, H. P., Morris, J. C. and Wakeham, Helmut.
DIFFUSION PHENOMENA IN SOLVENT EXTRACTION OF PEANUT OIL.
Ind. Eng. Chem 40, 195-199 (1948).
The theory of diffusion extraction of oil from a porous solid is examined in the light of previous investigations on solvent extraction of oilseeds. Peanut kernels were prepared and extracted in such a manner as to meet the conditions required by Fick's law. The diffusion coefficient under these conditions varies with solvent and with the moisture content of the oilseeds, but is essentially independent of the thickness of the peanut sections extracted. Results with peanut sections follow the theory closely when the broken cells at the surfaces, and the void spaces due to moisture loss, are taken into consideration. The techniques and conclusions presented may be of assistance in studying extraction from other oilseed systems.

Feuge, R. O. and Gros, Audrey T.
MODIFICATION OF VEGETABLE OILS. IX. PURIFICATION OF TECHNICAL MONOGLYCERIDES.
J. Am. Oil Chemists' Soc. 27, 117-122 (1950).

Technical monoglycerides containing 38.9% monoglycerides were purified by distributing them between two immiscible liquid phases (commercial hexane and an aqueous solution of methanol or ethanol). Products containing as much as 80% of monoglycerides were obtained in this manner. On fractionally crystallizing a solution of the same technical monoglyceride from aqueous ethanol, a glyceride fraction having a purity of 92% and containing 74% of the monoglycerides in the technical product was obtained.

Singleton, W. S.
PHASE INVESTIGATIONS OF FATS. II. SYSTEMS CONTAINING OLEIC AND STEARIC ACIDS AND AN ORGANIC SOLVENT.
J. Am. Oil Chemists' Soc. 25, 15-20 (1948).

Purpose of the investigation reported was to determine the composition of the solid and liquid phases existing in equilibrium in the ternary systems oleic acid-stearic acid-acetone, and oleic acid-stearic acid-commercial hexane, at various temperatures, and with varying amounts of the three components. Twelve ternary phase diagrams, constructed from compositional data of the liquid and solid phases in equilibrium at 0°C., -10°C., -20°C., -30°C., and -40°C., make possible prediction of the degree of separation which can be obtained with any given mixture of oleic and stearic acids, using either acetone or commercial hexane as solvent.

The intersolubilizing effect of oleic acid on stearic acid, greater in commercial hexane than in acetone, and the possible formation of mixed crystals of oleic and stearic acid were noted. Oleic acid of high purity can be obtained as one of the practical applications of these data.

Singleton, W. S.
PHASE INVESTIGATIONS OF FATS. III. SYSTEMS CONTAINING OLEIC AND PALMITIC ACIDS AND AN ORGANIC SOLVENT.
J. Am. Oil Chemists' Soc. 26, 332-336 (1949).

The ternary systems, oleic acid-palmitic acid-commercial hexane and oleic acid-palmitic acid-acetone, containing varying amounts of the three components were equilibrated at 0 degrees, -10 degrees, -20 degrees, -30 degrees, and -40 degrees C. From compositional data of the liquid and solid phases in equilibrium at each isotherm, ternary phase diagrams were constructed. From these diagrams, which are reproduced in the article, it is possible to predict the degree of separation which can be obtained with any given mixture of oleic and palmitic acids, using either acetone or commercial hexane as solvent. The intersolubilizing effect of oleic acid on palmitic acid was found to be greater in commercial hexane than in acetone, although each acid is more soluble in acetone than in commercial hexane,

Singleton, W. S. and Bailey, A. E.
TOCOPHEROL CONCENTRATES BY THE FRACTIONAL CRYSTALLIZATION OF COTTONSEED OIL FROM SOLVENTS.
Oil & Soap 21, 224-226 (1944).

Tocopherol concentrates were obtained from cottonseed oils by hydrogenating the oil and removing the bulk of the glycerides and sterols by low temperature crystallization from acetone. Completely hydrogenated oils are the best sources of concentrates at temperatures down to -60°C.; below this temperature partially hydrogenated oils are equally as good. A solvent-oil ratio of 8:1 by weight appears to be the optimum. Petroleum naphtha and methyl ethyl ketone are less suitable solvents than acetone, because of their greater capacity for dissolving glycerides at low temperatures.

Singleton, W. S., Lambou, Madeline and Bailey, A. E.
MODIFICATION OF VEGETABLE OILS. III. FRACTIONAL CRYSTALLIZATION OF FATTY ACIDS FROM SOLVENTS - SEPARATION OF THE SOLID AND LIQUID ACIDS OF COTTONSEED OIL.
Oil & Soap 22, 168-174 (1945).

An investigation is reported of low-temperature crystallization from organic solvents as a means of effecting practical separations of the solid and liquid acids of unhydrogenated and hydrogenated cottonseed oils. In the case of the hydrogenated acids, the solid acids include iso-oleic acids produced during the course of hydrogenation. At any fixed temperature the most efficient separations were obtained in the highly polar solvents, acetone and methyl acetate.

Skau, Evald L., Dopp, W. N., Burleigh, E. G. and Banowetz, L. F.
PHASE RELATIONS PERTAINING TO THE SOLVENT WINTERIZATION OF COTTONSEED AND PEANUT OILS IN ACETONE.
J. Am. Oil Chemists' Soc. 27, 556-564 (1950).

Systematic physical chemical data on the solvent-winterization behavior of cottonseed and peanut oils with acetone have been obtained which should serve as a basis for selecting the conditions necessary for the effective solvent winterization of these oils in acetone. The two oils are only partially miscible with acetone below certain temperatures which have been determined. In peanut oil this phenomenon may interfere with the winterization process within a certain range of concentrations, but for cottonseed oil the separation into two liquid phases does not occur until some 5°C. below the temperature required for adequate winterization. Complete data for a 3-hour holding-time have been obtained for three cottonseed oils ranging in iodine value from 106.1 to 116.4. Tables and graphs have been constructed to show the effect of oil-solvent ratio, chilling temperature, holding-time, agitation, and iodine value of the original oil on the percentage of solid removed and on the degree of winterization and iodine value of the winterized oil. Similar data have been obtained for a refined peanut oil insofar as possible without interference from separation into two liquid phases. It seems probable that if acetone were used as the winterization solvent for peanut oil, the separation into two liquid layers and the sensitivity of this phenomenon to moisture might be a source of processing difficulties especially if filtration instead of centrifugation were used to separate the solid from the supernatant.

SPECTRAL PROPERTIES

Boatner, Charlotte H., Hall, Catherine M., O'Connor, Robert T., Castillon, Leah E. and Curet, Maizie C.

PROCESSING OF COTTONSEED. I. PIGMENT DISTRIBUTION IN OILS AND MEALS PRODUCED BY HYDRAULIC AND SCREW PRESS METHODS.

J. Am. Oil Chemists' Soc. 24, 97-106 (1947).

An investigation of the changes which occur in gossypol and other pigments during the cooking and subsequent pressing of cottonseed by the hydraulic and continuous screw press methods was conducted in two parts: (1) laboratory-scale experiments on the effect of cooking; (2) mill-scale experiments on the effect of cooking and pressing. The pigmentation of cooked cottonseed was shown to depend upon the moisture content and period of heating of the seed. Crude hydraulic-pressed and screw-pressed oils produced under known processing conditions differed markedly from each other with respect to their original colors and refining characteristics. The screw-pressed crude oils were more deeply colored and contained one principal pigment, whereas the hydraulic-pressed oils contained two principal pigments. The absence of significant amounts of gossypol in the crude oils was demonstrated by means of a new technique for the quantitative isolation of gossypol. The crude oil pigments differed from gossypol, but like gossypol, were removed during alkali refining. The pigmentation of the crude oils has been shown to depend principally upon the pigmentation of the original seed and the moisture content of the seed during cooking. On the basis of absorption spectra, it appears that the alkali-refined hydraulic-pressed oils contain two to three pigments originally present in the crude oils whereas the alkali-refined screw-pressed oils contain in addition to these pigments a large number of decomposition products of the principal crude-oil pigment.

Boatner, C. H., Hall, C. M., O'Connor, R. T., Castillon, L. E. and Curet, M. C.

PROCESSING OF COTTONSEED. II. FACTORS DETERMINING THE DISTRIBUTION AND PROPERTIES OF PIGMENTS IN PRODUCTS PREPARED BY SOLVENT EXTRACTION.

J. Am. Oil Chemists' Soc 24, 276-283 (1947).

Criteria have been established which provide a basis for obtaining cottonseed oil and meal whose pigmentation may be more or less controlled. For the extraction of oil free of gossypol and gossypurpurin, moisture is excluded from the system and solvents other than water-miscible alcohols, ketones, and ethers are used. An alternative method is extraction of oil containing essentially all of the pigments of the seed by use of a number of water-miscible or other solvents capable of rupturing the glands. The pigment content of the solvent-extracted meals will be determined by the original pigment content of the seed, and the solubility of the pigments in the solvent employed. A third alternative is use of a special gland flotation process which in a single operation gives intact pigment glands and both oil and meal substantially free of pigments.

Budowski, Pierre, O'Connor, R. T. and Field, E. T.
SESAME OIL. IV. DETERMINATION OF FREE AND BOUND SESAMOL.
J. Am. Oil Chemists' Soc. 27, 307-310 (1950).

A spectrophotometric method for the determination of free and bound sesamol, based on the Villavecchia test, is described. Furfural, in the presence of aqueous sulfuric acid, proved the most suitable reagent for applying the Villavecchia test. It was possible to distinguish between free and bound sesamol by dissolving a sample of oil iso-octane; extracting with a dilute solution of aqueous alcoholic alkali in which the free, but not the bound, sesamol is soluble; and then applying the modified Villavecchia test to the original oil solution, the alkali-extracted oil solution, and the alkaline extract. The intensities of the colors produced with the three solutions correspond to the total, bound, and free sesamol, respectively, that was originally present in the oil. Agreement between the total and the free plus the bound sesamol in crude, refined, bleached, and deodorized samples of oil was close. Four crude oils of different origins had total sesamol ranging from 0.13 to 0.17 percent of which nearly all was bound. Yields of added sesamol varied from 95 to 106 percent. The method has proved useful in studies of the stability of sesame oil.

Budowski, Pierre, O'Connor, R. T. and Field, E. T.
SESAME OIL. VI. DETERMINATION OF SESAMIN.
J. Am. Oil Chemists' Soc. 28, 51-54 (1951).

A new method for the determination of sesamin in sesame oils is based on the measurement of the ultraviolet absorption of sesame oil following alkali isomerization and correcting for the presence of sesamolin. The new method's advantages are simplicity, accuracy, and lack of hazards. Ultraviolet absorption curves are given for sesamin, sesamolin, sesamol, and sesame oil.

Feuge, R. O., Pepper, M. B., Jr., O'Connor, R. T. and Field, Elsie T.
MODIFICATION OF VEGETABLE OILS. XI. THE FORMATION OF TRANS ISOMERS DURING THE HYDROGENATION OF METHYL OLEATE AND TRIOLEIN.
J. Am. Oil Chemists' Soc. 28, 420-426 (1951).

Infrared spectrophotometric technique for determining trans-octadecenoic acids and esters was applied to the hydrogenation of methyl oleate and triolein. The formation of trans isomers in the methyl ester was accelerated by increasing the temperature, increasing the catalyst concentration, and decreasing the degree of dispersion of hydrogen. Equilibrium was reached at 67% concentration of trans-isomers in the case of the methyl oleate, and less than 67% in oleic acid glyceride.

Fisher, G. S., O'Connor, R. T. and Dollear, F. G.
FATTY ACID COMPOSITION OF HYDROGENATED VEGETABLE OILS.
J. Am. Oil Chemists' Soc. 24, 382-387 (1947).

The fatty acid compositions of a number of unhydrogenated and hydrogenated peanut, cottonseed, soybean, and linseed oils have been calculated from the iodine number, thiocyanogen number, ultraviolet absorption after alkali-isomerization, and the content of saturated acids determined by a modified Bertram oxidation method. All the methods gave values in good agreement for the various samples of peanut oil. The iodine-thiocyanogen method and the modified Bertram oxidation method gave values in good accord for cottonseed oils; but the spectrophotometric method gave values for linoleic acid too high in the unhydrogenated and slightly hydrogenated cottonseed oils. Isolinoleic acid absorbs about 2 moles of thiocyanogen per mole of acid. With the spectrophotometric method for the determination of linolenic acid, the characteristic structure of the ultraviolet absorption band in the region of 250-275 mμ requires examination before attributing it to the isomerization of linolenic acid.

Graff, Morris M., and Skau, Evald L.
COLORED CHROMATOGRAMS WITH HIGHER FATTY ACIDS.
Ind. Eng. Chem., Anal. Ed. 15, 340-341 (1943).

A method is described for separating mixtures of higher fatty acids by Tswett adsorption analysis, whereby separation into zones was observed on a column of heavy magnesium oxide impregnated with a suitable indicator. The fatty acids were recovered by dissolving the magnesium oxide in acid and extracting with ether. It was possible to separate an unsaturated fatty acid from a saturated fatty acid of the same number of carbon atoms and of two saturated fatty acids differing in chain length by four carbon atoms.

O'Connor, Robert T., Field, Elsie T., Jefferson, M. E. and Dollear, F. G.
THE INFLUENCE OF PROCESSING ON THE SPECTRAL PROPERTIES OF VEGETABLE OILS.
J. Am. Oil Chemists' Soc. 26, 710-718 (1949).

Data are presented on the results of a detailed examination of the absorption spectra from 220 mμ to 720 mμ of typical cottonseed, soybean, peanut, sesame, okraseed, and rice bran oils. The absorption measurements were made on both crude and processed oils at successive processing steps of refining, bleaching, and deodorising. Findings are summarized for the ultraviolet region from 220 , for the near ultraviolet and the blue to blue-green portions of the visible region between 320 mμ and 500 mμ, and for the visible portion of the spectra above 500 mμ.

O'Connor, Robert T., Field, Elsie T. and Singleton, W. Sidney
THE INFRARED SPECTRA OF SATURATED FATTY ACIDS WITH EVEN NUMBER OF CARBON ATOMS FROM CAPROIC, C_6 (HEXANOIC), TO STEARIC, C_{18} (OCTADECANOIC), AND OF THEIR METHYL AND ETHYL ESTERS.
J. Am. Oil Chemists' Soc. 28, 154-160 (1951).

Data are plotted as percentage transmission for each compound against the wave length in μ . The exact wave length positions for the maximum of the eleven most prominent bands and correlations of each with molecular structure are given. A discussion of distinguishing acids from esters, the nonappearance of absorption due to either free or bonded OH group, and to association of the esters is given.

O'Connor, R. T., Heinzelman, D. C., Caravella, Maizie and Bauer, S. T.
SPECTRAL PROPERTIES OF LINOLEIC AND LINOLENIC ACIDS PREPARED BY DEBROMINATION.
Oil & Soap 23, 5-9 (1946).

Linoleic and linolenic acids were separated and purified by modifications of generally accepted methods. The adsorption spectra were determined for each of the acids which were obtained by the various modifications applied in their preparation. Extinction curves of the four linoleic acids and the two linolenic acids show successively decreasing absorption owing to the removal of or repression of conjugated impurities by progressively modified and improved methods of purification. The absorption spectrum of the product from each method of preparation was measured in the ultraviolet region from 220 to 320 $m\mu$, with a Beckman quartz spectrophotometer. The curve for linoleic acid prepared from corn oil showed marked absorption in the regions of diene conjugation, 232 $m\mu$, and of triene conjugation, 268 $m\mu$, and also measurable absorption in the tetraene region, 310 $m\mu$. Another curve for the above acid shows the effect of molecular distillation in its purification. Diene conjugated impurities were almost eliminated and triene and tetraene constituents were greatly reduced. Two other curves shows the continual reduction in conjugated components. Two similar curves are given for the ultraviolet absorption of linolenic acid. The data obtained offer an example of the utility of the spectrophotometer in following the progress of the preparation of a pure compound.

O'Connor, R. T., Heinzelman, D. C. and Dollear, F. G.
SPECTROPHOTOMETRIC ESTIMATION OF SOYBEAN OIL IN ADMIXTURE WITH COTTONSEED AND PEANUT OILS.
Oil & Soap 22, 257-263 (1945).

A spectrophotometric method is described for the determination of soybean oil in admixture with cottonseed oil that permits an accurate determination of linolenic acid for use as a criterion of the economic value of an oil mixture and as a guide in oil processing. The precision of the method is limited by variation in composition of the cottonseed and soybean oils in the mixtures. The proposed method can be extended to determine soybean oil in admixture with peanut or any other vegetable oil with negligible linolenic acid content.

O'Connor, R. T., Heinzelman, D. C., Freeman, A. F. and Pack, F. C.
THE SPECTROPHOTOMETRIC DETERMINATION OF ALPHA-ELEOSTEARIC ACID IN FRESHLY EXTRACTED TUNG OIL.
Ind. Eng. Chem. Anal. Ed. 17, 467-470 (1945).

The procedure for estimating alpha-eleostearic acid in freshly extracted tung oil by direct spectrophotometric measurements is studied and extinction coefficients for the pure acid in oil solvents are reported. A possible explanation of the discrepancies in the value of this coefficient in ethyl alcohol - i.e., partial deterioration during storage - has been given some confirmation by storage experiments. The extinction coefficients in hydrocarbon solvents have been used to determine the alpha-eleostearic acid content of some tung oil samples. The spectrophotometric procedure used is more direct, much simpler, and considerably more rapid than chemical methods.

O'Connor, R. T., Heinzelman, D. C. and Jefferson, M. E.
APPLICATION OF THE LINE-WIDTH METHOD TO THE SPECTROCHEMICAL ANALYSIS OF OILS, FATS, AND RELATED SUBSTANCES.
J. Am. Oil Chemists' Soc. 25, 408-414 (1948).

The line-width method applied to the spectrochemical analysis of oils and fats, in conjunction with an improved ashing technique, permitted quantitative determination by the authors of copper, iron, manganese, nickel, and tin in quantities as low as 1 part in 10 million. The procedure was critically examined by precision tests on commercial and experimental samples, by accuracy tests on synthetic samples, and by recovery tests. Results of actual analysis of 30 vegetable oils and fats indicate its usefulness as a research tool, especially in trace element analyses of organic materials that are low in ash content.

O'Connor, R. T., Heinzelman, D. C. and Jefferson, M. E.
PREPARATION OF ASH AND SPECTROCHEMICAL DETERMINATION OF TRACES OF METALLIC ELEMENTS IN OILS, FATS, AND RELATED SUBSTANCES.
J. Am. Oil Chemists' Soc. 24, 185-189 (1947).

Because even extremely small concentrations of any metallic elements present in fats or oils may affect quality, a special procedure of ashing fat or oil samples for spectrochemical analysis was developed. It is a modification of a magnesium nitrate ashing method and requires initial charring and drying _in vacuo_ followed by ashing in a furnace overnight. Traces of certain metals in ash obtained by this method can be detected when present in only a few parts per million. The preparation of the standards needed to obtain the calibration curves is discussed. Using given spectrum lines, germanium meets the requirements for an internal standard for the determination of aluminum, copper, iron, nickel, and manganese; and lead, for tin and zinc. The use of direct current gives the best results. A rotating sector having four steps can be used to enable all lines to be measured at suitable densities to permit either reading of the intensities from straight line portions of the plate calibration curve, or measurement of the lines of the element to be determined and of the internal standard at equal densities — depending upon which of two generally used methods is applied to obtain the metallic concentration. The accuracy of the method appears to be within the variations encountered in successive determinations of a metal in the same sample of oil. Spectrograms of typical vegetable oils are shown.

O'Connor, Robert T., Heinzelman, Dorothy C., McKinney, R. S. and Pack, F. C.

THE SPECTROPHOTOMETRIC DETERMINATION OF THE ALPHA AND BETA ISOMERS OF ELEOSTEARIC ACID IN TUNG OIL.

J. Am. Oil Chemists' Soc. 24, 212-216 (1947).

An application of the well-known method for the spectrophotometric analysis of binary systems to the simultaneous determination of the alpha and beta isomers of eleostearic acid in tung oil is described. Complete absorption curves of the two pure isomers and of two tung oil samples in cyclohexane show that the absorption differences between the two isomers are not very great. The results obtained with use of the method on a selected group of tung oils — including (1) fresh oils containing relatively large proportions of alpha eleostearic acid, (2) irradiated oils with large proportions of the beta isomer, (3) stored samples of oil with intermediate amounts of both alpha and beta isomers, and (4) oils obtained by different methods of extraction — are compared with results obtained by the use of chemical methods for the determination of total eleostearic acid based on maleic anhydride values. As a test of the reliability of the procedure, the absorption spectra of tung oil and of mixtures of pure alpha and beta eleostearic acids in cyclohexane were compared. A further check was provided by adding the determined values of the two isomers and comparing this result with an independently determined value for the total eleostearic content. The results indicated that the proposed method offers a reasonably accurate and rapid means for determining alpha, beta, and total eleostearic acids in all types of tung oils.

O'Connor, R. T., Poninski, C. H., Heinzelman, D. C., Howell, Hilda and Von der Haar, Patricia.

INFLUENCE OF CONDITION OF SEED ON THE SPECTRAL PROPERTIES OF CRUDE AND PROCESSED HYDRAULIC COTTONSEED OILS.

J. Am. Oil Chemists' Soc. 29, 220-224 (1952)

The influence of condition and of storage of cottonseed on the spectral properties of the crude oils obtained by hot and cold hydraulic pressing and of refined, bleached, and deodorized oils obtained from them are reported. The spectral data are presented as curves of the logarithm of the extinction coefficients as the ordinate and the wavelength as the abscissa. The intensities of the absorption bands at several characteristic wavelengths are discussed.

O'Connor, R. T., Stansbury, M. F., Damaré, H. G. and Stark, S. M., Jr.

A STUDY OF THE SPECTROPHOTOMETRIC METHOD FOR POLYUNSATURATED FATTY ACIDS IN COTTONSEED OILS AND A COMPARISON WITH CHEMICAL METHODS.

J. Am. Oil Chemists' Soc. 29, 461-466 (1952).

Forty-eight cottonseed oils, selected to represent a random distribution with respect to variety, station, year of growth, and iodine value, have been analyzed for fatty acid content by the spectrophotometric method. Equations for calculating the linoleic acid content from spectrophotometric data have been simplified. The spectrophotometrically determined values for linoleic acid are compared with chemically determined values, and found to differ with an average of 2.4%, with the spectrophotometric values being uniformly higher.

Planck, Ralph W., Pack, Frank C. and Heinzelman, Dorothy C.
STABILITY OF ELEOSTEARIC ACIDS DURING STORAGE.
Address, Am. Tung Oil Assoc. Proc., Oct. 11-13, 1950.

Alpha- and beta-eleostearic acids prepared from tung oil were stored under various conditions and the amounts of decomposition were determined by spectrophotometric examination. The results of alpha-eleostearic acid gave exposure to atmosphere at -4° to 35°C. as causing viscous decomposition products; it is less stable than the beta-acid. Storage of the alpha-acid below -40° resulted in less decomposition. Beta-eleostearic acid stored in the form of a paste with ethanol in filled and stoppered containers was quite stable.

Pons, Walter A., Jr., Murray, Mildred D., O'Connor, Robert T. and Guthrie, John D.
STORAGE OF COTTONSEED AND PEANUTS UNDER CONDITIONS WHICH MINIMIZE SPECTROPHOTOMETRIC CHANGES IN THE EXTRACTED OIL.
J. Am. Oil Chemists' Soc. 25, 308-313 (1948).

The work reported in this paper was undertaken in an effort to establish new indexes of deterioration and to further verify previous recommendations for the storage of cottonseed and peanuts. The increase in the seed of red gossypol (a term used to designate substances exhibiting absorption maxima in chloroform at 528 and 565 mμ), and also increased absorption of the extracted oil in the region of 236 mμ, proved helpful in following changes in condition during storage of the seed. Methods for applying both these indexes are described.

Cottonseed with moisture content adjusted to 8 percent or less, stored for more than a year in closed containers at 1 degree or at -18 degrees C. (conditions under which previous experiments indicated cottonseed and peanuts may be stored with no increase in free fatty acid content of the oil) showed no appreciable change in either red gossypol content of the seed, or nongossypol absorption of the extracted oil; while seed stored at room temperature, 27 degrees C., showed a large increase in both. Oil extracted from peanuts stored for more than four years at room temperature showed a much greater absorption in the region of 227 to 234 mμ than oil from peanuts stored at the lower temperatures.

Spadaro, J. J., McCourtney, E. J. and Vix, H. L. E.
SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. VII. EFFECT OF DRYING FLAKED PRIME COTTONSEED ON COLOR OF OIL AND MEAL PROPERTIES.
J. Am. Oil Chemists' Soc. 27, 394-396 (1950).

In an investigation of the effect of the heat in drying cottonseed flakes in preparation for solvent extraction in oil color and meal properties, prime cottonseed from the 1948 crop of New Roads, Louisiana, was used. The seed, which had been stored under favorable conditions, had 6. percent initial moisture content and 1.2 percent free fatty acid content. The results showed that, although the colors increased slightly with increased drying temperatures of the flakes, all of the oil colors were prime. This indicates that dark oils produced by solvent extraction from this lot of cottonseed are due to conditions other than flake drying temperatures.

Swift, C. E., Mann, G. E. and Fisher, G. S.
GAMMA-TOCOPHEROL AS A PRECURSOR OF A RED QUINOID SUBSTANCE DEVELOPED IN COTTONSEED OIL DURING OXIDATION.
Oil & Soap 21, 317-320 (1944).

On the basis of spectrophotometric data on the characteristic spectral absorptions observed in oxidizing substrates with and without added γ -tocopherol, it is shown that the red color which develops on accelerated oxidation of fat substrates containing this tocopherol results from its conversion to chroman-5,6-quinone. The chroman-5,6-quinone thus produced possesses relatively little antioxidant activity.

Williams, P. A., Boatner, C. H., Hall, C. M., O'Connor, R. T. and Castillon, L. E.
PROCESSING OF COTTONSEED. III. COLOR DEVELOPMENT IN COTTONSEED OIL DURING STORAGE OF THE SEED AND CRUDE OIL.
J. Am. Oil Chemists' Soc. 24, 362-369 (1947).

Processing of cottonseed by the hydraulic press method has been carried out at two mills, one located about 100 miles farther south than the other. The varieties of seed processed and conditions during processing, which included cooking of moistened seed, were essentially the same at both mills. Seeds were also processed at the more southerly mill by the screw press method for which seed was cooked without added moisture. The crude hydraulic- and screw-pressed oils produced were stored at different temperatures for 10 months, and samples were periodically refined and bleached. Seeds were also stored at the two mills and were periodically processed to permit comparison between the characteristics of oils from stored seed and those of stored oils. The absorption spectra of the crude, refined, and bleached oils were determined. The rate of increase of bleach color in the oils during storage of the seed and crude oils has been correlated with temperature of storage and changes in the absorption spectra of the oils.

Williams, P. A., Hadden, R. P., Hall, C. M., Castillon, L. E., Guice, W. A., O'Connor, R. T. and Boatner, C. H.
PROCESSING OF COTTONSEED. IV. EFFECT OF PREPARATION AND COOKING OF MEATS ON THE BLEACH COLOR AND STORAGE PROPERTIES OF SCREW-PRESSED OILS.
J. Am. Oil Chemists' Soc. 26, 28-34 (1949).

In order to study their effect on the characteristics of cottonseed meal and oil produced by screw-pressing, conditions of processing were varied with respect to preparation of meats, addition of water, and the temperature and duration of cooking. In laboratory-scale experiments 10 percent of water added to the flaked meats prior to cooking at 235 to 244 degrees F. for 1-1/2 hours resulted in a low bleach color in the expressed oils. Mill-scale tests indicated that cooking in the presence of relatively large amounts of water improves the oil's storage properties but not its initial bleach color. On the other hand, unwetted rolled or ground meats cooked at a low temperature resulted in low initial bleach color of the oil with very little reversion during storage at room temperature. Widely varying amounts of gossypol were found in both oils and meals produced from meats cooked under various conditions.

THERMAL PROPERTIES

Bailey, A. E., Jefferson, M. E., Kreeger, F. B. and Bauer, S. T.
THERMAL PROPERTIES OF FATS AND OILS. IV. SOME OBSERVATIONS ON THE POLYMORPHISM AND X-RAY DIFFRACTION CHARACTERISTICS OF TRISTEARIN AND A HIGHLY HYDROGENATED COTTONSEED OIL.
Oil & Soap 22, 10-13 (1945).

Melting points and x-ray diffraction patterns are determined for cottonseed oil hydrogenated to an iodine value of less than 1 and for highly purified tristearin. The results for the two materials are compared and point to a well-defined crystal structure, hitherto unreported, for both materials in the lower melting forms. The patterns of the two materials differed in the highest melting forms. A new pattern was observed for tristearin at the intermediate melting point. Four polymorphic forms of the hydrogenated cottonseed oil were detected.

Bailey, A. E. and Oliver, G. D.
THERMAL PROPERTIES OF FATS AND OILS. III. CALORIMETRIC ESTIMATION OF SOLID AND LIQUID GLYCERIDES IN COTTONSEED OIL AND HYDROGENATED COTTONSEED OIL.
Oil & Soap 21, 300-302 (1944).

The relative amounts of solid and liquid glycerides in cottonseed oil and a hydrogenated cottonseed oil have been estimated calorimetrically at different temperatures over the melting ranges of the oils. A correlation is established between the content of solids in hydrogenated cottonseed oil and the consistency of the oil, as measured by micropenetrations.

Charbonnet, Grace H. and Singleton, W. S.
THERMAL PROPERTIES OF FATS AND OILS. VI. HEAT CAPACITY, HEATS OF FUSION AND TRANSITION, AND ENTROPY OF TRILAURIN, TRIMYRISTIN, TRIPALMITIN, AND TRISTEARIN.
J. Am. Oil Chemists' Soc. 24, 140-142 (1947).

Heat capacities of the *a*- and *b*-forms of trimyristin, tripalmitin, and tristearin, and the *b*-form of trilaurin were measured. The heats of fusion of the *b*-forms of these four compounds were determined. Those of the *a*-forms of trimyristin, tripalmitin, and tristearin were calculated from heat content data. Calculations were also made of the heats of transition, *a*- to *b*-form. The molal entropy at 298.16°K. was calculated for the *b*-form of each compound.

D'Aquin, E. L., Spadaro, J. J., Vix, H. L. E., Pominski, J., Molaison, L. J. and Pollard, E. F.
SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. III. THE EFFECT OF HEAT ON COTTONSEED OIL IN MISCELLAS FROM UNCOOKED AND COOKED FLAKED MEATS.
Oil Mill Gazetteer 51, 17-19 (1947).

Crude oils produced on a pilot-plant scale under comparable conditions from a single lot of prime cottonseed by hexane extraction of uncooked and cooked meats and by standard hydraulic pressing of the cooked meats were compared as to quality. The crude oils were then subjected to various heating temperatures for 1 hour to simulate evaporative processing in commercial oil and solvent recovery, and the relative effect of heating, as indicated by the color of the oils after caustic refining and bleaching with Fuller's earth, was judged. All three oils were of prime grade and were comparable in refining loss, refined color, and bleached color. The oil extracted from the uncooked meats had a lower refining loss and a lighter color than either of the other two oils. In all cases the bleached oils were darker in color as the heating temperature was raised. The oil from the uncooked meats appeared to degrade in color more rapidly at the 150° and 240°F. temperature levels than did the oil from the cooked meats; and its final bleached color was darker after heating at 240° F. The hydraulic-pressed oil was markedly less affected by heating than either of the hexane-extracted oils and did not exhibit a sharp bleached color degradation as did the latter.

Decossas, Kenneth M., Mackey, Harvey A. and Heughan, Gordon P.
BOILING POINTS OF COTTONSEED AND PEANUT OIL MISCELLAS IN ENGLISH UNITS.
AIC-257 (Processed) (1950).

Existing data on boiling point - vapor pressure - constant composition parameters for crude cottonseed oil - and for crude peanut oil-commercial hexane miscellas are replotted in English units; namely, in inches of mercury vacuum and in degrees of temperature Fahrenheit.

Morgan, D. A.
SMOKE, FIRE, AND FLASH POINTS OF COTTONSEED, PEANUT AND OTHER VEGETABLE OILS.
Oil & Soap 19, 193-198 (1942).

The smoke points of good quality cottonseed and peanut oils differ but little. The presence of free fatty acids in these oils lowers the smoke point for (1) these refined oils have an increase in smoke point after bleaching with no change in free acid content; (2) prolonged heating of these oils causes an initial increase followed by a decrease in smoke point with increasing fatty acid content; and (3) the smoke point decreases at a more rapid rate on long heating than can be accounted for by the rate of increase of free acid. Highly hydrogenated oils do not follow this pattern on heating, and it would appear that the smoke point is lowered by unsaturated materials, such as carotenoid pigments.

Oliver, G. D. and Bailey, A. E.
THERMAL PROPERTIES OF FATS AND OILS. V. THE HEAT CAPACITY AND HEAT OF FUSION OF HIGHLY HYDROGENATED COTTONSEED OIL.
Oil & Soap 22, 39-41 (1945).

The heat content of a quickly chilled and of a slowly chilled and tempered sample of almost completely hydrogenated cottonseed oil was measured over a temperature range within which there was in each case complete transformation of the oil from a solid to a liquid form. Heat capacity data for the liquid oil and for the quickly chilled and the tempered solid oil were calculated. Equations expressing the changes in heat capacity with temperature were derived. A correlation of the heat capacity data on highly hydrogenated cottonseed oil with similar data previously obtained on unhydrogenated and on partially hydrogenated cottonseed oil is shown. The heat of fusion calculated for both the quickly chilled and the tempered solid oil is given.

Oliver, G. D., Singleton, W. S., Todd, S. S. and Bailey, A. E.
THERMAL PROPERTIES OF FATS AND OILS. II. HEAT CAPACITY AND LATENT HEAT OF COTTONSEED OIL.
Oil & Soap 21, 297-300 (1944).
The heat content, specific heat in both solid and liquid states, and heat of fusion of cottonseed oil, a hydrogenated cottonseed oil, and a mixture of cottonseed oil and petroleum naphtha are presented. Equations are presented for expressing the heat capacities of the oils and the oil-solvent mixture with the oil in either the solid or liquid form.

Pollard, E. F., Vix, H. L. E. and Gastrock, E. A.
SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. BOILING POINT-VAPOR PRESSURE-COMPOSITION RELATIONS FOR MISCELLAS OF OILS IN HEXANE.
Ind. Eng. Chem. 37, 1022-1026 (1945).
The boiling points and densities of mixtures of cottonseed and peanut oils with commercial hexane are reported. They are useful in the design of vacuum evaporators and strippers and for control operations involving temperature, time of heating, and concentration of oil-solvent mixtures of various compositions, to prevent or minimize fixation of objectionable coloring matter or other deteriorative heat effects. Boiling-point data are determined at various concentrations of crude cottonseed oil and crude peanut oil, over a range of pressures from 160 to 760 mm. absolute in commercial hexane. Observations of the effect of agitation in establishing equilibrium conditions of the oil-solvent mixtures are noted.

Vix, H. L. E., Pollard, E. F., Spadaro, J. J. and Gastrock, E. A.
SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OIL. EFFECT OF HEAT ON COTTONSEED OIL MISCELLAS.
Ind. Eng. Chem. 38, 635-642 (1946).
This investigation is a practical effort to evaluate the effect of temperature and time of heating during processing operations upon the fixation of objectionable color in miscella obtained by extraction of uncooked cottonseed flakes with commercial hexane. The results reported were obtained from work on one lot of prime cottonseed. Color fixation became objectionable between 150° and 180° F., and beyond 180° increased rapidly. Properly prepared solvent-extracted cottonseed oils from a prime lot of seed were successfully refined by

slightly modified official A.O.C.S. refining methods which yielded oils of prime quality with low refining losses. The finished oils were equal in quality to high-grade hydraulic-pressed oils. Results have yielded necessary engineering information useful for design and operation of pilot plant equipment and for processing operations. Absorption spectra curves ranging through the visible and ultraviolet are reproduced as examples of applicability of this technique for studying solvent-extracted cottonseed oils. Operating data for heating, as well as complete refining data are tabulated.

Ward, T. L. and Singleton, W. S.
THERMAL PROPERTIES OF FATS AND OILS. VII. HYDROGENATED AND UNHYDROGENATED PEANUT OILS.
J. Am. Oil Chemists' Soc. 27, 423-426 (1950).

A refined and bleached peanut oil was examined calorimetrically before and after hydrogenation. Measurements were made over the entire range of melting, from completely solid to liquid. Values for the specific heats of the oils were used to develop equations relating temperature and specific heat of the oils in both solid and liquid states. The heat of fusion of both samples was also determined. The relative amounts of solid and liquid glycerides in hydrogenated and unhydrogenated peanut oil at various temperatures over their entire melting ranges were estimated from the calorimetric data.

Ward, T. L., Singleton, W. S. and Planck, R. W.
THERMAL PROPERTIES OF FATS AND OILS. VIII. SPECIFIC HEATS, HEATS OF FUSION, AND ENTROPY OF ALPHA AND BETA TUNG OILS.
J. Am. Oil Chemists' Soc. 29, 155-157 (1952).

Beta tung oil was found capable of existing in three polymorphic forms, melting at 52.8°, 44°, and 28°C., respectively. Equations were developed to express the specific heats of the stable forms of tung oil. The specific heat of beta tung oil was found to be higher when the sample was rapidly cooled than when it was slowly cooled or tempered. The heat of fusion of beta oil was 16.12 cals./g., and the alpha oil, 21.02 cals./g. Entropies were 231.04 and 252.15 at 298.16°K., respectively. The liquid glycerides present at any temperature in the melting range were estimated from the heat content of the oils.

VISCOSITY AND DENSITY

Bauer, S. T. and Gastinel, V., Jr.
EFFECT OF METALLIC SALTS ON THE STABILITY OF PALM AND HYDROGENATED COTTONSEED OILS.
Oil & Soap 21, 36-42 (1944).

The relative effects of added metallic compounds upon the formation of free fatty acids and the rate of increase in the viscosity of heated palm and hydrogenated cottonseed oils are reported. It was found that when these oils were heated in the presence of tin to temperatures comparable with those obtaining in the tin pot during the hot dipping of tin, palm oil showed a slightly lower rate of increase in viscosity compared to the cottonseed oil. Ferric oxide had a marked effect on the rate of viscosity increase of palm oil, but produced no change in the rate of increase in viscosity of the cottonseed oil. Polymerization of both oils as measured by the rate of increase in viscosity, was retarded by the addition of zinc salts. The addition of ferric oxide or ferric stearate accelerated the rate of viscosity increase.

Decossas, Kenneth M., Deckbar, Frederick A., Jr. and Hecker, Joseph L.
DENSITIES AND GRAVITIES OF COTTONSEED AND PEANUT OIL MISCELLAS IN ENGLISH UNITS.
AIC-292 (Processed) (1950).

Data in the literature on density-temperature-constant composition parameters for refined and winterized cottonseed oil-commercial hexane miscellas and for refined winterized peanut oil-commercial hexane miscellas are replotted in English Units; namely, densities in pounds per cubic foot and per gallon, and temperatures in degrees Fahrenheit. The specific gravities are calculated on a basis of the density of water at 60°F. These plots complement those previously published (see list for January-June 1950, Item No. 12) in English units on boiling points of cottonseed and peanut oil miscellas. Replotting these data will render them more useful and suitable for design calculations, plant operations, and interpretations of operating data.

Decossas, Kenneth M., Deckbar, Frederick A., Jr. and Hecker, Joseph L.
VISCOSITIES OF COTTONSEED AND PEANUT OIL-HEXANE MISCELLAS IN ENGLISH UNITS.
AIC-304 (Processed) (1951).

Existing data on viscosity-temperature curves for crude cottonseed oil- and for crude peanut oil-commercial hexane miscellas are replotted in English units.

Magne, Frank C., Huges, Edwin J. and Skau, Evald L.
DENSITY-COMPOSITION DATA FOR COTTONSEED OIL-SOLVENT MIXTURES.
J. Am. Oil Chemists' Soc. 27, 552-555 (1950).

Density-composition-temperature data are reported for binary systems of a refined cottonseed oil with methyl pentane, diethyl ether, trichloroethylene, and tetrachloroethylene over a wide temperature range -- from the point of incipient phase separation to within 10° to 15°C. of the boiling point of the solvent. These data can be used to calculate the density knowing the composition and temperature or, conversely, the composition knowing the density and the temperature. An equation has been derived which makes it possible to adjust these data so that they apply with reasonable accuracy to other refined and crude cottonseed oils and even to soybean or peanut oil systems with the same solvents.

Magne, F. C. and Skau, E. L.
VISCOSITIES AND DENSITIES OF SOLVENT-VEGETABLE OIL MIXTURES.
Ind. Eng. Chem. 37, 1097-1101 (1945).

A pycnometer and a viscometer suitable for use with volatile mixtures and for low-temperature determinations are described. Density and viscosity measurements are made from incipient crystallization to a temperature near boiling point of the solvent for the complete binary systems of cottonseed oil with Skellysolve B (commercial hexane); cottonseed oil with acetone; cottonseed oil with 2-butanone; peanut oil with Skellysolve B; and soybean oil with Skellysolve B. Density- and viscosity-composition curves for the binary systems are shown and discussed.

Magne, Frank C. and Wakeham, Helmut.
VISCOSITIES AND DENSITIES OF HYDROGENATED PEANUT OILS.
Oil & Soap 21, 347-349 (1944).

The melting point, solidification point, viscosity, and density of different varieties of peanut oil and hydrogenated peanut oil are reported. A plot of the densities of hydrogenated peanut oil against the iodine values at several temperatures gives a family of isotherms from which densities of hypothetical oils of known iodine value can be read. An equation relating density and temperature for a peanut oil of given iodine value is found to be

$$d_2 = d_1 - 0.000646\ (t_2 - t_1)$$

A plot of the viscosities against temperatures for peanut oils of different iodine values gives a family of isotherms from which viscosities of hypothetical oils of known iodine value can be determined. Viscosity and temperature of a fatty oil is related by the equation

$$\log\log\ (v + 0.8) = m(\log T_1 - \log T) + \log\log\ (v_1 + 0.8)$$

where v and v_1 are kinematic viscosities at absolute temperatures T and T_1, respectively.

Wakeham, Helmut and Magne, Frank C.
VISCOSITIES AND DENSITIES OF HYDROGENATED COTTONSEED OILS.
Ind. Eng. Chem. 36, 563-570 (1944).

The viscosity and density of different varieties of cottonseed oils and hydrogenated cottonseed oils are reported. A plot of the densities of hydrogenated cottonseed oil against the iodine values at several temperatures gives a family of isotherms from which densities of hypothetical oils of known iodine values can be read. An equation relating density and temperature for a cottonseed oil of given iodine value is found to be

$$d_2 = d_1 - 0.000638(t_2 - t_1)$$

A plot of the viscosities against temperatures for cottonseed oils of different iodine values gives a family of isotherms from which viscosities of hypothetical oils of known iodine value can be determined.

MISCELLANEOUS PHYSICAL PROPERTIES

Andraos, Victor, Swift, C. E. and Dollear, F. G.
SESAME OIL. I. PROPERTIES OF A SOLVENT-EXTRACTED SESAME OIL.
J. Am. Oil Chemists' Soc. 27, 31-34 (1950).

Data are presented with respect to the extraction, processing characteristics, and the chemical and physical characteristics of oil obtained by extraction from white sesame seed. Progressive changes occurring in the composition, stability, plasticity, and refractive index of the fat during selective hydrogenation of the refined and bleached oils are presented.

Bailey, A. E. and Fisher, G. S.
MODIFICATION OF VEGETABLE OILS. V. RELATIVE REACTIVITIES TOWARD HYDROGENATION OF THE MONO-, DI-, AND TRIETHANOID ACIDS IN CERTAIN OILS.
Oil & Soap 23, 14-18 (1946).

From the analysis of several series of hydrogenated cottonseed, soybean, and linseed oils, made by the spectral and other methods, estimates have been made of the relative readiness of hydrogenation of oleic, iso-oleic, linoleic, and linolenic acids, and an isomer of linoleic acid which presumably has its double bonds in the 9:10 and 15:16 positions. Under selective conditions of hydrogenation the relative reactivities of the unsaturated fatty acids toward hydrogen may be approximately represented by the following whole numbers: oleic acid 1; isooleic acid 1; isolinoleic acid 3; linoleic acid 20; linolenic acid 40. Use of "nonselective" conditions reduces the reactivity of linoleic and linolenic acids as a group in comparison to that of oleic and isolinoleic acids as a group but does not appreciably alter the relative reactivities of the two acids within the same group. In nonselective hydrogenation the ratio of reactivity of linoleic acid to that of oleic acid may be as low as 5, as compared with 20 or above under selective conditions. The hydrogenation of linoleic acid to stearic acid appears to proceed substantially in two stages, with intermediate desorption of oleic acid from the catalyst, but there is evidence that the hydrogenation of linolenic acid may in part proceed directly to oleic acid, without intermediate desorption of linoleic acid. In both selective and nonselective hydrogenation, the hydrogenation of linolenic acid to linoleic acid and to isolinoleic acid appears to proceed at approximately equal rates.

Bauer, Stewart T. and Markley, K. S.
HYDROGENATED COTTONSEED OIL AS A SUBSTITUTE FOR PALM OIL IN THE PRODUCTION OF TIN PLATE AND COLD REDUCED SHEET STEEL.
Oil & Soap 20, 1-11 (1943).

Work is reported on the development of a substitute for palm oil, used in the cold reduction process for the manufacture of strip steel. Included are the chemical and physical properties of virgin and spent palm oils, and the effect of these properties on the action of these oils in the tin bath. It was found that a selectively hydrogenated cottonseed oil with an iodine value of about 50 was best adapted as a substitute for palm oil. The effect of heating on the viscosity, rates of free acid formation, effect of flux, and other factors are determined.

Bickford, W. G., Mann, G. E. and Markley, K. S.
POSSIBILITIES OF PEANUT, PECAN, AND SAFFLOWER SEED OILS AS SUPPLEMENTS FOR OLIVE OIL.
Oil & Soap 20, 85-89 (1943).
Samples of various oils were examined for spectral transmittance, composition, refractive index, smoke, flash, and other properties, as possible supplements for olive oil. None of the oils examined simulate olive oil in composition, although peanut and pecan oils appear capable of modification to produce a product similar to olive oil and for certain purposes can replace olive oil without modification.

Budowski, Pierre
SESAME OIL. VII. OPTICAL ROTATION AND THE MINOR COMPONENTS OF SESAME OIL.
J. Am. Oil Chemists' Soc. 28, 54-55 (1951).
The optical rotations of four crude sesame oils are calculated on the basis of their known contents of sesamin, sesamolin, and phytosterols and the reported optical activities for these components. The optical rotations of the sesame oils are shown to be the result of sesamin, sesamolin, and phytosterol content.

Feuge, R. O. and Janssen, H. J.
BLEACHING OF COTTONSEED OIL IN HEXANE.
J. Am. Oil Chemists' Soc. 28, 429-432 (1951).
Results are presented for the batch-bleaching of 30% solutions of cottonseed oil-commercial hexane. With activated clay and carbon, reduction in color was greater in the presence of solvents, other conditions being equal. Countercurrent bleaching of the miscella in absorbent columns are highly efficient with respect to utilization of the absorptive capacity of clay, and to amount of oil loss by absorption.

Feuge, R O. and Reddi, P. B. V.
RICE BRAN OIL. III. UTILIZATION AS AN EDIBLE OIL.
J. Am. Oil Chemists' Soc. 26, 349-353 (1949).
An odorless, tasteless, and neutral product well suited for use as a cooking oil was obtained by conventional refining, bleaching, and deodorization of rice bran oil produced on a pilot plant scale. The smoke, flash, and fire points were comparable to those of other high quality edible oils. Rice bran oil solidifies less easily than either cottonseed or peanut oil, is more resistant to oxidative rancidity than are the average cooking oils, and can be winterized with little difficulty. During hydrogenation rice bran oil behaves like a typical vegetable oil.

Hussain, S. A. and Dollear, F. G.
CHARACTERISTICS OF SOLVENT-EXTRACTED AND HYDRAULIC-PRESSED OKRASEED OILS.
J. Am. Oil Chemists' Soc. 27, 295-300 (1950).

The production, characteristics, composition, and stability of solvent-(commercial hexane-) extracted hydraulic-pressed okraseed oils and their hydrogenated products were compared. The okraseed was of the Louisiana Green Velvet variety, grown in Louisiana in 1947. Solvent-extracted okraseed oil gave a very low refining loss and the refined and bleached oil was light-colored oil (10 yellow and 1.0 red, bleached with 4 percent acid-activated earth). The hydraulic-pressed oil had a higher refining loss, a higher free fatty acid content, and when refined and bleached the lowest color obtained was 35 yellow and 4.3 red Lovibond units. After deodorization the color was 35 yellow and 2.2 red. The two oils were hydrogenated to different iodine values at 15 p.s.i. pressure, 250°F., with 0.1 percent nickel catalyst. The calculated composition based on spectrophotometric measurements was not in agreement with that calculated from iodine and thiocyanogen values; however, repeated isomerizations and measurements gave fairly good agreement with values obtained by chemical methods. Stabilities of the refined, bleached, and hydrogenated oils were somewhat lower than those of cottonseed or peanut oils of comparable iodine values. The studies indicate that solvent-extracted and hydraulic-pressed okraseed oil can be used in edible products.

Hussain, S. A., Dollear, F. G. and O'Connor, R. T.
OIL FROM THE KERNELS OF LALOB FRUIT, *BALANITES AEGYPTIACA*.
J. Am. Oil Chemists' Soc. 26, 730-732 (1949).

Lalob fruit from the Sudan was examined by modern chemical and physical methods. The kernels, which amount to 9 to 10 percent of the fruit, contain approximately 46 percent oil. Extraction of the ground kernels with commercial hexane yields a light yellow oil, having a composition calculated from iodine-thiocyanogen values corresponding to 43.8 percent linolein, 30.5 percent olein, 23.7 percent saturated acid glycerides, and 2.0 percent unsaponifiable matter. Calculation of the composition from ultraviolet absorption measurements gave results in fairly close agreement with those obtained by the iodine-thiocyanogen method. It was shown that the color of the crude oil was due almost entirely to the presence of *a*-carotene.

Kyame, Lillian, Fisher, G. S. and Bickford, W. G.
HYDRAZIDES OF *n*-ALIPHATIC ACIDS.
J. Am. Oil Chemists' Soc. 24, 332-334 (1947).

The hydrazides of the normal fatty acid series from valeric through lauric as well as those of myristic, palmitic, and stearic acids have been prepared, and their melting points and mixed melting points have been determined. All of the products are crystalline solids at room temperature and have melting points between 62° and 116°C. The melting point differential, approximately 10°, between the lowest adjacent members decreases as the series is ascended, being only 3° between palmitic and stearic acid. A plot of the melting points of the hydrazides vs number of carbon atoms gave a smooth curve exhibiting no alternation between even and odd members of the series. The hydrazides are readily prepared from the corresponding esters and appear to be satisfactory derivatives for identifying fatty acids of moderate molecular weight.

Menezes, F. G. T., Budowski, Pierre and Dollear, F. G.
SESAME OIL. II. SOME CHEMICAL AND PHYSICAL PROPERTIES OF THE OILS FROM DIFFERENT VARIETIES OF SESAME SEED.
J. Am. Oil Chemists' Soc. 27, 184-186 (1950).

Four varieties of sesame seed were solvent extracted and the oils examined with respect to their physical and chemical characteristics. The oils were refined, bleached, hydrogenated to shortening consistency, and deodorized. Changes in composition and in values of the refractive index and plasticity during hydrogenation were similar to those of other oils of similar characteristics. Changes in constants and fatty acid composition during hydrogenation are essentially similar to those occurring in other vegetable oils such as cottonseed or peanut oils.

Murti, K. S. and Dollear, F. G.
RICE BRAN OIL. II. COMPOSITION OF OIL OBTAINED BY SOLVENT EXTRACTION.
J. Am. Oil Chemists' Soc. 25, 211-213 (1948).

Interest in the recovery of oil from rice bran produced by southern and southwestern mills, and the lack of data on the characteristics and composition of such oil, promoted an analysis of three samples --- two of crude oil and one of refined oil. These oils were obtained by solvent-extraction of commercial rice brans from Texas-grown Blue Bonnet and Arkansas-grown Zenith varieties of rice from the 1946 crop. Methods used in making the analysis are outlined, and the results summarized both in tabular and discussion form. Briefly, the glyceride composition of the samples was found to be: 0.80, 1.06 and 0.84 percent linolenic acid; 33.2, 30.6 and 33.1 percent linoleic acid; 45.0, 46.0 and 46.3 percent oleic acid; 17.1, 17.3 and 17.1 percent saturated acids; and 3.9, 5.0 and 2.7 percent unsaponifiable matter, respectively.

Reddi, P. B. V., Murti, K. S. and Feuge, R. O.
RICE BRAN OIL. I. OIL OBTAINED BY SOLVENT EXTRACTION.
J. Am. Oil Chemists' Soc. 25, 206-211 (1948).

Freshly milled rice bran of good quality was extracted with commercial hexane, yielding an oil of relatively low free fatty acid content, with good color, and as stable as other similar types of crude oils. When the temperature was kept below 10°C. and the extraction discontinued at the right time, the recovered oil represented 90 to 95 percent of the total lipids in the bran. It contained very little wax -- about 3 to 9 percent of the total extractable lipids -- and this could be extracted easily with hot commercial hexane and other solvents. It was found that good rice bran oils behave under ordinary refining methods much like cottonseed oils of comparable free fatty acid content. Losses approximating the absolute or Wesson loss resulted from caustic soda refining in a hydrocarbon solvent as well as from refining with sodium carbonate. When bleached in the usual way, some of the refined rice bran oils produced products acceptable for use in the edible trade. The greenish cast caused by the presence of chlorophyll was removed by bleaching with a small amount of activated clay.

Shahani, H. S., Dollear, F. G. and Markley, K. S.
THE BUFFALO GOURD, A POTENTIAL OILSEED CROP OF THE SOUTHWEST DRYLANDS.
J. Am. Oil Chemists' Soc. 28, 90-95 (1951).
The analysis of the seed and solvent-extracted meal, with respect to moisture, protein, fiber, and ash of the buffalo gourd is given. Also, oil color, refractive index, composition, etc., is reported. The color of the processed oil was inferior to that of other common edible oils.

Smith, B. A. and Dollear, F. G.
OIL FROM CALABASH SEED, CRESCENTIA CUJETE L.
J. Am. Oil Chemists' Soc. 24, 52-54 (1947).
The composition and characteristics of calabash, *Crescentia cujete* L. seed and oil have been determined. The oil was found to have the following composition calculated from the iodine and thiocyanogen values and saturated acid content of mixed fatty acids: saturated acids 19.7%, oleic acid 59.4%, linoleic acid 19.3%, and linolenic acid 1.6%. Comparison of the composition and characteristics of calabash seed oil with those of peanut and olive oils indicates that, except for the small amount of linolenic acid present in the former, the oils are similar.

Soltoft, P. and Dollear, F. G.
EVALUATION OF SAFFLOWERSEED OIL IN EDIBLE FAT PRODUCTS.
J. Am. Oil Chemists' Soc. 28, 335-338 (1951).
The compositions of the seed, hulls, meal, and oil of four varieties of seed are reported. The refining, hydrogenation, and deodorization processing tests indicate that the oil is not as suitable for edible purposes as other domestic edible oils.

Swift, C. E., Dollear, F. G. and O'Connor, R. T.
THE OXIDATION OF METHYL OLEATE. I. THE PREPARATION, PROPERTIES AND REACTIONS OF METHYL HYDROPEROXIDO OLEATE.
Oil & Soap 23, 355-359 (1946).
The oxidation of the methyl ester of oleic acid, one of the principal acid components of the glycerides of cottonseed oil, was investigated. A method was devised for the preparation and isolation of the first product of oxidation of methyl oleate, methyl hydroperoxido oleate. Information was obtained relative to the structure and the chemical and physical properties of the hydroperoxide. The hydroperoxide was inactive towards *alpha*-tocopherol when the two compounds were mixed and stored at room temperature.